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Journal of the European Ceramic Society 23 (2003) 1223–1231

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Processing optimization and mechanical evaluation of hot pressed 2D Tyranno-SA/SiC composites

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Received 26 October 2001; received in revised form 20 August 2002; accepted 30 August 2002

Abstract

Non-coated and carbon-coated 2D woven Tyranno SA/SiC composites were fabricated by hot pressing (HP) via liquid phase sintering (LPS). Various preparation conditions were optimized using non-coated fiber fabrics and mechanical evaluation was further conducted for the composites using carbon coated fiber fabrics. Results have indicated that intra-bundle infiltration of the fiber preforms during polymer impregnation and pyrolysis (PIP) pretreatment would affect the density of the composites. Modification of the polymer (polycarbosilane—PCS) to filler (SiC with sintering additives) ratio could promote the densification process, especially the intra-bundle matrix formation. Using non-coated fiber preforms, at 1750 °C, 15MPa, suitable polymer precursor and filler content [PCS/(PCS + SiC) = 50%] was beneficial for obtaining composites with higher density. Applying carbon coated fiber fabrics and adopting the optimized polymer to filler ratio for pretreatment, composites with non-catastrophic fracture behavior could be obtained. Meanwhile, carbon coating can well protect fiber from damaging. At 1780 °C under 20 MPa, higher density of 2.82 g/cm^3 could be reached. This composite exhibited a relatively strong fiber/matrix bonding and the improved mechanical properties such as ultimate strength, proportional limit stress and elastic modulus, either in bending or tensile tests.

Keywords: Densification; Fracture; Hot pressing; Mechanical properties; SiC/SiC

1. Introduction

SiC/SiC composites are well-known materials promising for high temperature structural applications because of their intrinsic properties. Previous studies have mainly concentrated on the lower temperature fabrication methods such as chemical vapor infiltration (CVI),[1–5] polymer impregnation and pyrolysis (PIP)[6,7] and reaction sintering (RS).[8,9] By modification of the interfacial properties (using fiber coating) and the infiltration techniques, these preparation techniques have provided high performances to SiC/SiC composites.

Hot pressing is an effective processing technique to densify powder compacts, especially in the case of inclusion of reinforcement,[10,11] because at elevated temperature, pressure is simultaneously applied to promote the densification process. Since the potential damage to fiber reinforcement exists, very little has been done on SiC/SiC composite fabrication using this technique. The high oxygen content for the earlier developed SiC based fibers such as Nicalon fiber are thermodynamically unstable so that their application for SiC/SiC composite fabrication is limited because of the decomposition of the Si–O–C phase.[12–14] Meanwhile, the fibers with low crystallinity like Hi-Nicalon still cannot tolerate high pressure heat treatment at high temperature even though the oxygen content is rather low because it might cause creep deformation leading to the degradation in strength.[15]

With the development of advanced fibers, well-crystallized and near-stoichiometric SiC fibers like Tyranno SA fibers have been achieved.[16,17] This fiber has high tensile strength and modulus and shows no degradation in strength or change in composition on heating to 1900 °C in an inert atmosphere and in air at 1000 °C. The elimination of the oxygen and perfect crystallization in this fiber makes it possible to fabricate SiC/SiC composites at a relatively high temperature and in harsh conditions such as hot pressing. However, potential fiber degradation at high temperature and high

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PII: S0955-2219(02)00298-4

pressure might affect the effectiveness for fiber reinforcement.

The purpose of this paper is to optimize preparation conditions for the densification process and evaluate the mechanical performances of SiC/SiC composites fabricated by hot pressing via liquid phase sintering using Tyranno SA as reinforcement. The effects of the densification process on the mechanical behavior for the composites using carbon coated fiber fabric reinforcement are also discussed.

2. Experimental procedure

2D woven Tyranno SA fiber fabrics (Ube Industries Ltd., Japan) were used as the reinforcement to fabricate SiC/SiC composites. Processing condition optimization was mainly conducted using non-coated fiber fabrics and further composite fabrication was conducted using carbon-coated fiber preforms. The thickness of the carbon (PyC) coating is about 0.3 μm. To lower the sintering temperature, β-SiC nano-power (Marketech International Inc., America) was used for the matrix formation. Typical composition of this nano-powder is as follows: SiC > 95%, Free C 1–2%, O 1–1.5%, and the average particle size is below 30 nm. Al_2O_3 (Sumitomo Chemical Industries Ltd., Japan) and Y_2O_3 (Johnson Matthey, UK) were used as sintering aids.

Fiber preforms were firstly impregnated by polymer precursor (polycarbosilane—PCS) with filler (β-SiC nano-power containing sintering additives of Al_2O_3 and Y_2O_3) at various compositions and then pyrolysed. These pretreated fiber preforms were cut into squares approximately 30 by 30 mm, which were ready for further composites preparation. β-SiC nano-power was mixed with Al_2O_3 and Y_2O_3 in an attritor ball miller for 3 h to form the slurry. The cut squares of fiber preforms were dipped into the mixed slurry and then moved out for drying. The dipping and drying process was repeated allowing a certain amount of solid matrix to adhere to the fiber preforms to form the inter-bundle matrix. Fiber volume fraction was controlled at about 35–40 vol.% depending on the sintering conditions of the composites. Those prepared preforms, which were coated with matrix, were stacked in a graphite die for hot pressing. Temperature was varied from 1720 to 1780 °C, while the pressure was changed from 15 to 20 MPa. For comparison, monolithic SiC was also prepared by hot pressing the dried matrix powder under an identical pressure of 15 MPa and the temperature was also changed from 1720 to 1780 °C.

The hot pressed samples were subsequently cut and ground into about 4 × 1.8 × 26 mm size for three-point bending test in an INSTRON 5581 test machine, with the crosshead speed of 0.5 mm/min and the span, 18 mm. Tensile test was also performed on some samples using carbon coated fiber fabrics as reinforcement. The size of the tensile specimens was 3 × 1.8 × 30 mm. On both ends of the tensile bars, aluminum tabs were affixed to each side using a standard Araldite binder. The gauge length was designated to be 15 mm. During the tensile test, cyclical unloading–reloading was applied on the tensile bars through the aluminum tabs on both ends of the samples, which were clamped on the fixture mounted on the test machine. Tensile strain was recorded from the extensometer adhered on both sides of the gauge areas.

Density of each sample was measured by Archimedes' method. Both the polished cross-section and the fracture surface after the bending and tensile tests were observed by scanning electron microscopy (SEM).

3. Results and discussion

3.1. Optimization of densification process using non-coated fiber fabrics

3.1.1. Effect of sintering conditions

For SiC/SiC composite fabrication by hot pressing, potential fiber degradation caused by high temperature (especially under pressure) needs to be considered. If the densification process could be effectively promoted, the sintering temperature and pressure should be as low as possible. It has been reported that by liquid phase sintering using Al_2O_3 and Y_2O_3 as additives, low temperature densification of monolithic SiC becomes possible.[18] However, using micrometer SiC powders, the densification process is still not efficiently achieved when the temperature is below 1800 °C. To lower the sintering temperature, SiC nano-powder was used at the present experiment and the composition of the sintering additives was carefully modified. The total amount of sintering additives is about 10 wt.%, including a small amount of SiO_2. Al_2O_3 to Y_2O_3 weight ratio is 3:2. The effect of the sintering conditions on the densification process both for monolithic SiC and SiC/SiC composites using non-coated fiber fabrics is shown in Fig. 1. It is clearly indicated that densification of the composites was retarded by the inclusion of fiber preforms into the matrix. For monolithic SiC, a highly densified compact could be obtained when temperature is over 1750 °C under 15 MPa. At 1780 °C, about 99% of the theoretical density could be reached. Although the densification was retarded by the inclusion of fiber reinforcement, high density of the composite could be obtained, from 2.9 g/cm^3 at 1750 °C to 2.95 g/cm^3 at 1780 °C.

Increasing pressure (over 15 MPa) could also improve the densification process, however, fiber deformation would occur in using non-coated fiber fabrics even at 1750 °C.

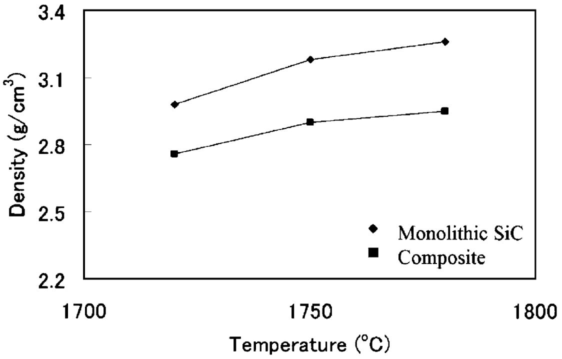

Fig. 1. The relationship between density and hot pressing temperature for monolithic SiC and SiC/SiC composites using non-coated fiber fabrics under the pressure of 15 MPa.

3.1.2. Effect of impregnation and pyrolysis pretreatment

To form the intra-bundle matrix and improve the density of the composite, a pre-impregnation and pyrolysis technique was adopted prior to the introduction of the fiber preforms into the powder compact. Various compositions of PCS/(PCS + SiC) were selected for modifying the impregnation efficiency. Fig. 2 shows the effects of polymer/filler composition on the densification process at 1750 °C under 15 MPa.

It can be identified that higher density (2.9 g/cm^3) could be obtained at PCS/(PCS + SiC) = 50%. When PCS/(PCS + SiC) = 30% and PCS/(PCS + SiC) = 100%, both of the composites have a slightly lower density. Usually, higher SiC content is beneficial for increasing the solid content in the impregnated and pyrolysed matrix to promote the densification effectively. However in the present experiment, since the nano-powder used has very high specific surface area (110 m^2/g), higher SiC addition would greatly increase the viscosity of the slurry. To fit for the intra-bundle infiltration, a relatively larger amount of solvent for dispersion was necessary so that the real solid content impregnated into the fiber bundles might be decreased. In this case, the infiltration efficiency is lowered and simultaneously retards the densification process. As the polymer content increases, solvent used for dispersion will be simultaneously decreased so that the infiltration efficiency is improved. In the case without filler addition [PCS/(PCS + SiC) = 100%], the total pyrolysed intra-bundle matrix (only from polymer precursor) might be even low so that densification process will be depressed. Actually, since the bulk contraction of the infiltrated slurry is extremely large for nano--powders, even at PCS/(PCS + SiC) = 50%), it might still not be enough to fill the intra-bundle vacancy perfectly.

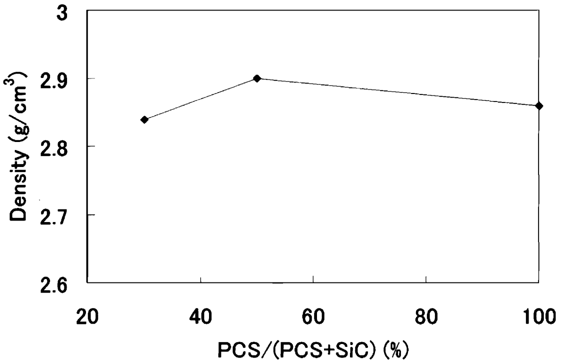

Fig. 2. Effect of polymer/filler composition on the densification process at 1750 °C, 15 MPa.

3.1.3. Microstructural evolution

The influence of the sintering temperature on the microstructural evolution is indicated in Fig. 3. As the sintering temperature is at 1720 °C, relatively loosely formed intra-bundle matrix could be identified. This behavior might be ascribed to the low ability for liquid phase formation at such a low temperature. In this case, sintering is not enough to be promoted. When the temperature was increased to 1750 °C, a well-densified intra-bundle matrix was revealed. At this temperature, the promotion of densification was well realized as indicated in Fig. 1. At 1780 °C, fibers were highly compacted and the intra-bundle matrix almost sintered together with the fibers. Comparison of the appearance of the fiber, an obvious characteristic could also be identified. At 1720 and 1750 °C, fibers almost keep their original feature–round in shape. While in the higher temperature (1780 °C) sintered composite, fiber deformation could be observed. This is probably because the high sinterability of the matrix at higher temperature leads to the enhanced contraction of the composite under pressure so that the applied pressure was greatly exerted on the contacted fibers leading to the fiber deformation.

Comparison of the composites hot pressed at 1750 °C, 15 MPa with different polymer and filler compositions is demonstrated in Fig. 4. No big difference could be distinguished with the variation of polymer to filler ratio. Some micropores exist in the intra-bundle areas in all the composites.

As explained in the previous section, intra-bundle matrix formation is highly dependent on the impregnation efficiency during the PIP pretreatment. The introduction of polymer precursor can improve either fiber bundle infiltration efficiency or the wettability between fibers and matrix slurry. However, for polymer precursor, the conversion of PCS to the amorphous pyrolysed products and then crystallization at high temperature will be accompanied by the large volume shrinkage. Combined with the sintering of nano-powder SiC with high specific surface area, pores will be left in the intra-bundle areas. During sintering (especially

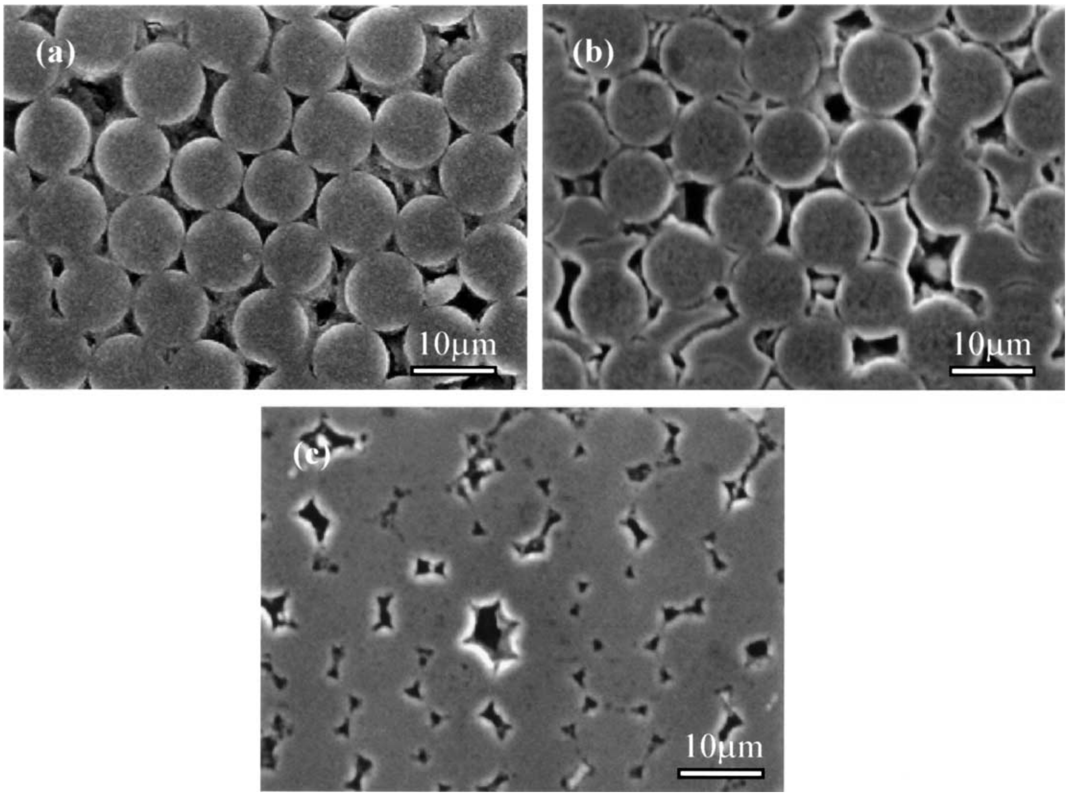

Fig. 3. Comparison of the microstructure on the polished cross-section. Composites were prepared under 15 MPa at different temperature: (a) 1720 °C, (b) 1750 °C, and (c) 1780 °C.

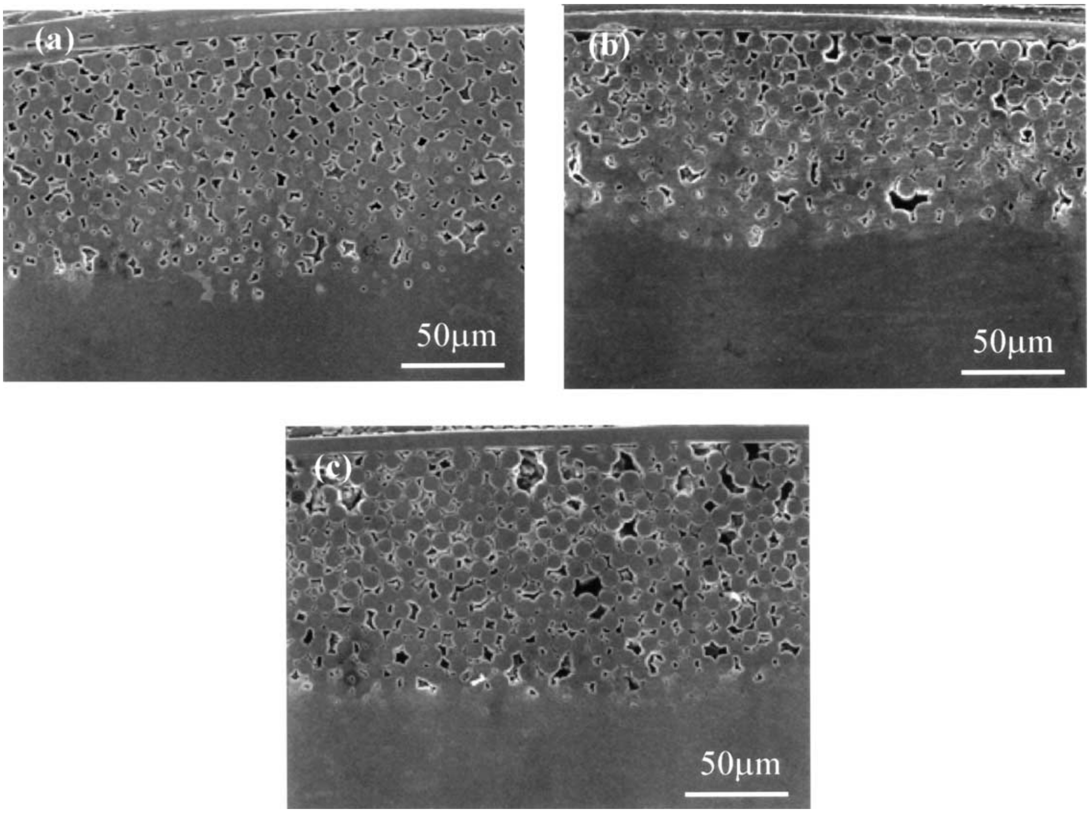

Fig. 4. SEM micrographs on the polished cross-section showing the composites prepared at 1750 °C, 15 MPa with different polymer and filler composition: (a) PCS/(PCS + SiC) = 100%, (b) PCS/(PCS + SiC) = 50%, (c) PCS/(PCS + SiC) = 30%.

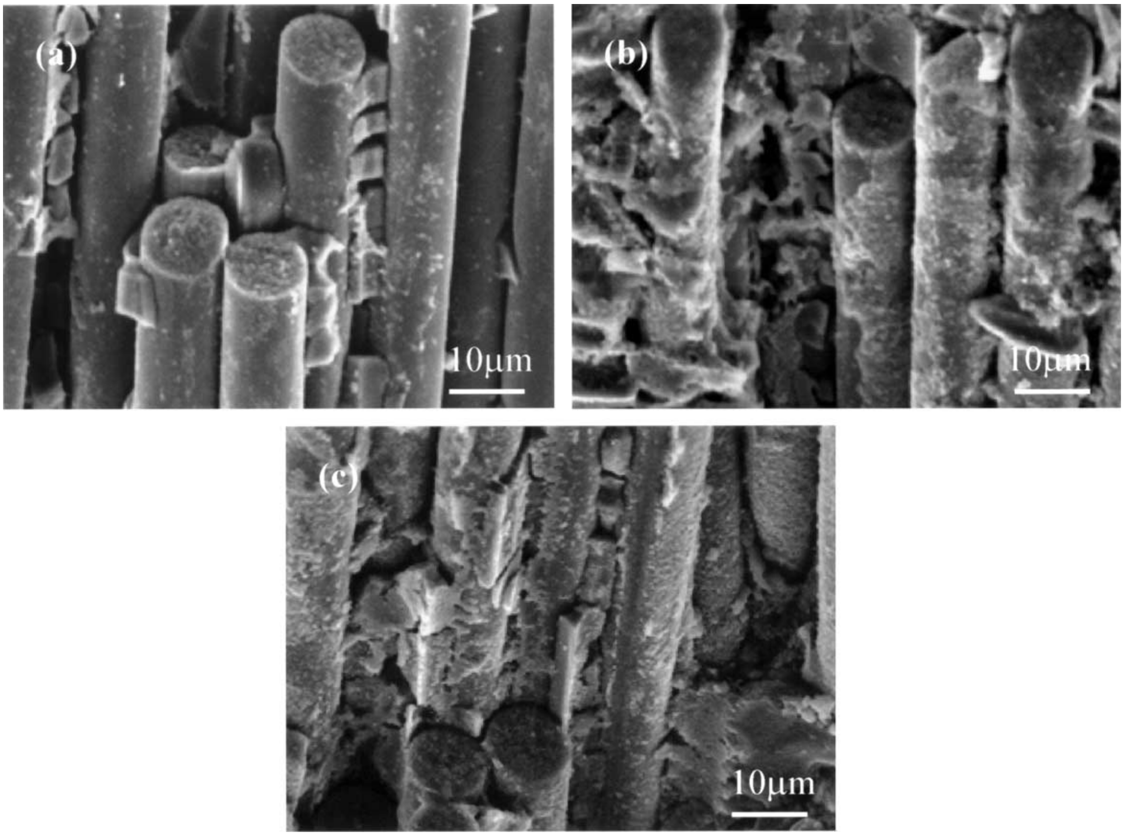

Fig. 5. SEM observation on the fracture surface of the composites hot pressed at 1750 °C, 15 MPa with different polymer and filler composition: (a) PCS/(PCS + SiC) = 100%, (b) PCS/(PCS + SiC) = 50%, (c) PCS/(PCS + SiC) = 30%.

under pressure), some of them would be removed by fiber rearrangement, but some would be still left.

To further understand the appearance of the intra-bundle matrix, the fracture surface was examined, as shown in Fig. 5. It could be observed that discontinuous intra-bundle matrix with cracks was formed at PCS/(PCS + SiC) = 100% (Fig. 5a). When filler was incorporated during fiber preform pretreatment, continuous matrix was revealed (Fig. 5b and c), indicating that the composed composition for fiber preform impregnation and pyrolysis was beneficial for intra-bundle matrix formation. These results are consistent with the previous explanation. Meanwhile, the interaction between matrix and fibers could also be identified from the later two micrographs.

3.2. Densification and evaluation of the composites using carbon coated fiber fabrics as reinforcement

3.2.1. Physical and mechanical properties

Since the sintering temperature and pressure, and polymer impregnation and pyrolysis pretreatment would affect the densification process, carefully modification of the densification conditions is necessary during composite fabrication. However, due to the strong bonding between fibers and matrix when no fiber coating was applied, the composites demonstrated brittle fracture behavior. Further experiment was conducted using carbon coated fiber fabrics with the optimized pretreatment condition [PCS/(PCS + SiC) = 50%]. Meanwhile, because of fiber coating, higher sintering

Table 1
Effects of experimental conditions on the physical and mechanical properties of the composites

Sintering conditions	1750 °C/15 MPa	1780 °C/15 MPa	1780 °C/20 MPa
Density (g/cm^3)	2.67	2.77	2.82
Porosity (%)	13.9	10.6	9.1
Bending strength (MPa)	267.6 ± 11.8	299.8 ± 19.1	331.4 ± 31.1
Proportional limit stress (MPa)	160.0 ± 21.2	207.5 ± 37.6	238.3 ± 18.9
Elastic modulus (GPa)	110.2 ± 7.0	115.0 ± 2.8	129.5 ± 3.8

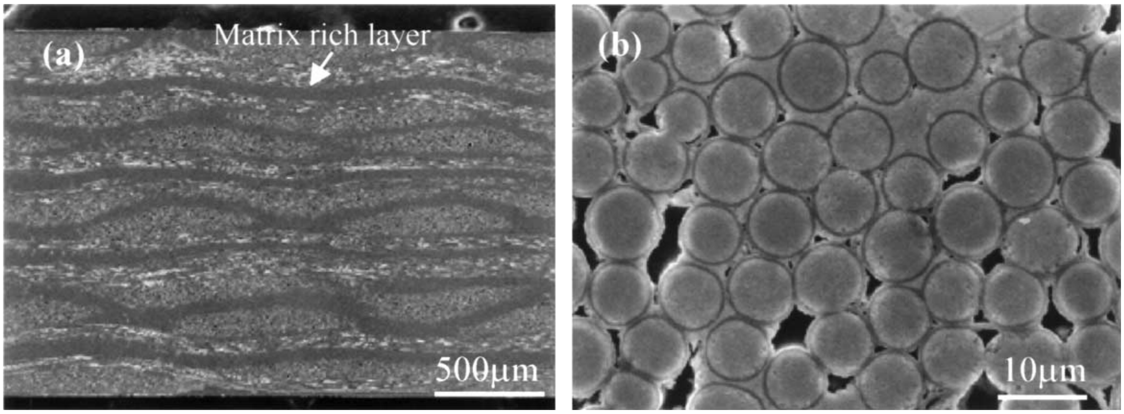

Fig. 6. SEM observation on the polished cross section for the composite using carbon-coated fiber fabrics and hot pressed at 1780 °C, 20 MPa showing the matrix layers formation (a) and intra-bundle micropores distribution (b).

temperature and pressure were also extended. Table 1 lists some physical and mechanical properties for the composites using carbon coated fiber fabrics. Although the inclusion of carbon coating will decrease the theoretical density of the composites, compared with the composite using non-coated fiber preforms at the similar sintering conditions, the density of the present composite is still at a lower level. It might be partly ascribed to the compatibility between matrix/fiber (SiC to SiC) and matrix/carbon (SiC to C). The former one would allow the fiber rearrangement easily during hot pressing and the later one might more or less prohibit the fiber rearrangement.

Increasing temperature (or simultaneously increasing pressure) will enhance the densification of the composites. Meanwhile, the mechanical properties are also improved. At 1780 °C, 20 MPa, relatively higher density with better mechanical performances were obtained. Even at these sintering conditions, only few fiber deformations could be detected, as shown in Fig. 6(b). This is quite different from the composite using non-coated fiber fabrics, indicating that the fiber coating can well protect fibers from damaging.

From Fig. 6(a), it can be clearly observed that the matrix rich layers were formed by the presently adopted fabrication technique. In this case, the densification of the composite would be actually divided to two parts: inter-bundle matrix densification and intra-bundle matrix densification. Inter-bundle matrix densification is similar to that of the monolithic SiC and it is a temperature dependent process, as shown in Fig. 1. Although the inclusion of fiber reinforcement would retard the densification process, at above 1750 °C under 15 MPa, highly densified inter-bundle matrix should be achieved. For intra-bundle matrix formation, since the PIP formed products would have some residual carbon during the pyrolysis of the PCS polymer precursor,[19] the sinterability would be more or less inhibited. Meanwhile, fibers rearrangement was also dependent on the sinterability of the intra-bundle matrix. At higher temperature and pressure such as at 1780 °C under 20 MPa, the liquid phase formation would more effectively promote the grain boundary sliding so that the fibers rearrangement would be more enhanced. It should be borne in mind that during this process, higher external pressure would simultaneously be imposed on the surface of the fibers, potential damage to fibers might occur with the increment of both temperature and pressure.

As illustrated in Fig. 6(b), many micropores still remained in the intra-bundle areas. This would be the reason that hinders the further improvement of the density.

3.2.2. Fracture behaviors

Fig. 7 shows the stress/displacement curves from the bending test for the composites using carbon coated fiber fabrics and hot pressed at different temperature and different pressure. These curves also reveal the different characteristics such as the ultimate bending strength, proportional limit stress and elastic modulus, and demonstrate the non-catastrophic fracture behavior. Comparison of these curves, highest values for mechanical properties are also obtained at 1780 °C

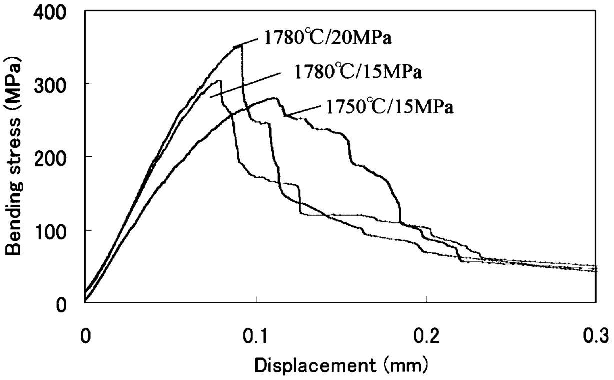

Fig. 7. Stress/displacement curves from bending test for the composites using carbon-coated fiber fabrics and hot pressed at different conditions.

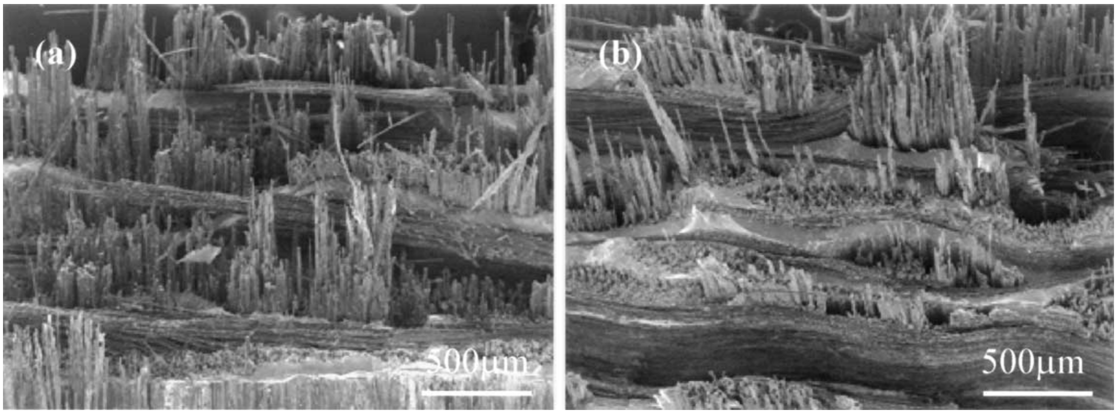

Fig. 8. Fracture surface from bending test showing the different characteristics for the composites sintered at different conditions: (a) 1750 °C, 15 MPa and (b) 1780 °C, 20 MPa.

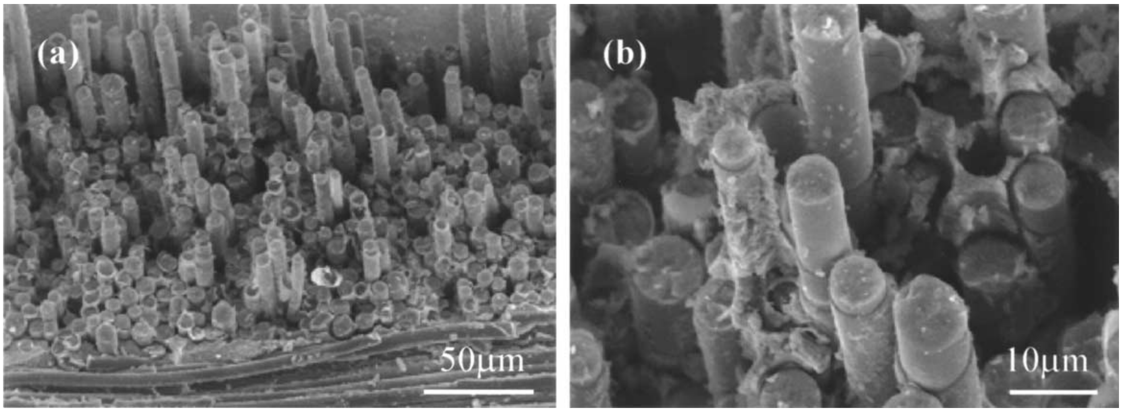

Fig. 9. SEM observation on the fracture surface from bending test showing the detailed information of the pulled out fibers for the composite sintered at 1780 °C under 20 MPa.

under 20 MPa, which are consistent with the results listed in Table 1.

Further comparison of the fracture surface for the composites prepared at 1750 °C under 15 MPa and 1780 °C under 20 MPa is indicated in Fig. 8. In the composite sintered at low temperature and low pressure (1750 °C under 15 MPa), relatively long fiber pull-out dominates the fracture surface. In this composite, carbon coating was mainly adhered on the fiber surface during fracture indicating that the matrix cracking occurred along the matrix/carbon interface. When sintering temperature and pressure increased to 1780 °C and 20 MPa, many of the pulled out fibers become short. In this composite, crack deflection happened either in the fiber/carbon interface or carbon/matrix interface, as evidenced in Fig. 9. This figure also demonstrates the typical fracture characteristics of the short fiber pull-out region.

The above features for each composite might be ascribed to the microstructural evolution at different sintering conditions. At low temperature with low pressure, lower densified matrix allows crack initiation and propagation easily. Meanwhile, a relatively weak fiber/matrix bonding is formed in this composite. All these features would be beneficial for long crack deflection so that the long fiber pull-out would occur. But in this case, it is simultaneously detrimental to strengthening. When both temperature and pressure are increased a relatively well-densified matrix would hinder crack initiation and arrest crack propagation, and simultaneously enhance fiber/matrix bonding. These characteristics would then lead to the short crack deflection and make the pulled out fiber become short. However, in this composite, relatively long fiber pull-out could still be observed, indicating that some weak fiber/matrix bonding exists allowing the long crack deflection. Actually, it can be comprehended from the Fig. 6(b), in which some fibers are surrounded by micropores to form the weak bonding regions. These regions would take precedence to allow crack initiation and propagation, which is similar to that of the low temperature with low pressure sintered composite.

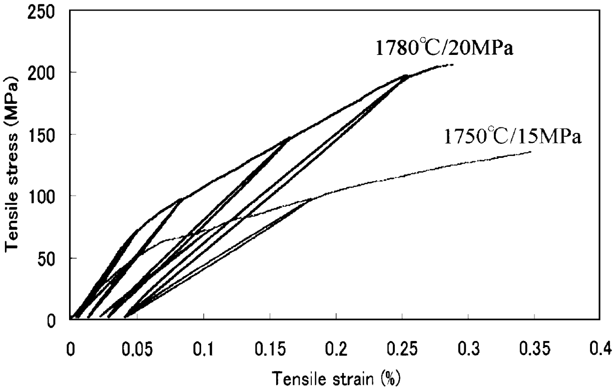

Fig. 10. Tensile stress–strain curves with unloading–reloading cycles for the composites sintered at different conditions: (a) 1750 °C, 15 MPa and (b) 1780 °C, 20 MPa.

Fig. 10 gives the tensile stress–strain curves with unloading–reloading cycles for the composites sintered at different conditions. An obvious characteristic is that these two composites also show the different slope of the initial linear region (implying the ability for elastic deformation), different proportional limit stress and different appearance for hysteresis loops. For the composite sintered at 1780 °C under 20 MPa , relative narrow hysteresis loops and decreased permanent strain at zero loads at the similar tensile stress suggest the stronger fiber/matrix bonding and the higher ability for load transfer from matrix to fibers.[20,21] From the continuous enhancement of the mechanical properties with the increment of temperature and/or pressure, fiber degradation might be limited by the fiber coating. However, since the intra-bundle matrix densification will finally affect the crack initiation and propagation, and fiber/matrix bonding, improvement of the intra-bundle matrix formation technique will be the important approach, and it will become the basis for future efforts.

4. Conclusions

2D woven Tyranno SA/SiC composites have been fabricated by hot pressing via liquid phase sintering. Non-coated fiber fabrics were used as reinforcement for processing optimization and carbon-coated fiber fabrics were further used as reinforcement for mechanical evaluation.

Using non-coated fiber fabrics, at 1750 °C, 15 MPa, suitable polymer precursor/filler content [PCS/(PCS + SiC) = 50%] was beneficial for obtaining the composite with higher density (~2.9 g/cm^3) and well consolidated intra-bundle matrix. In this case, either increasing temperature or pressure would lead to the deformation of fibers.

Using carbon-coated fiber fabrics, densification process would be retarded compared with that using non-coated fiber fabrics as reinforcement at the similar preparation conditions. Increasing temperature and pressure further promote the densification of the composites and enhance the mechanical performances. In this case, carbon coating could well protect fibers from damaging and all these composites demonstrated the non-catastrophic fracture behavior. For the lower temperature and pressure (1750 °C under 15 MPa) sintered composite, long fiber pull out dominates the fracture behavior and relatively weak fiber/matrix bonding was formed. At 1780 °C under 20 MPa, a higher density of 2.82 g/cm^3 could be obtained. This composite exhibited a fracture behavior mainly with short fiber pull-out and a relatively strong fiber/matrix bonding. Mechanical properties such as ultimate strength, proportional limit stress and elastic modulus, either from a bending test or tensile test, were simultaneously improved.

Acknowledgements

This work is supported by the CREST, Japan Science and Technology Corporation and conducted at the Institute of the Advanced Energy, Kyoto University.

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